



ANALYTICAL INSTRUMENTS GROUP

APD 2000 PRO

POWDER X-RAY DIFFRACTOMETER



www.gnr.it

About us

GNR, thanks to its 40 years of experience, is a worldwide market manufacturer of advanced analytical instruments, developing procedures of analysis for various applications, supplying the corresponding laboratory equipment and providing consulting and Customer support worldwide, through its well established sales and post-sale network.

GNR designs and manufactures Optical Emission Spectrometers (OES) and Rotating Disc Electrode Optical Emission Spectrometers (RDE-OES) for the measurement of elemental composition of metal alloys and the analysis of contaminants, additives and wear metals in oils and lubricants, coolants and hydraulic fluids.



GNR Head Office and Production Site is located in Agrate Conturbia (Novara), near Lago Maggiore; 20 minutes from MALPENSA Airport.



GNR designs and produces X-ray Diffractometers (XRD) and X-ray Fluorescence Spectrometers (XRF) for the study of material structure and elemental composition for both academic and industrial applications.

Certified Company

Highest quality in our products and services is a core value for **GNR**.

Full commitment is dedicated to support our quality system in the overall process and continuous improvement is fundamental to guarantee GNR compliance to the internationally accepted quality management standard ISO 9001.



GNR periodically organizes at its facility courses and training for technicians and agents as well as seminars and demonstrations.



Thanks to an extensive network of agents GNR provides technical support and delivers spare parts worldwide.

In relation to the process of continuous development, GNR reserves the right to change specifications of the instruments without previous notice at any time; the real ones will always be those shown in the final order confirmation.





GNR Analytical Instruments Group is a worldwide market leader in supplying advanced X-ray (XRD, XRF) and Optical Emission Spectrometer (OES) systems for complete solutions in structural and elemental analysis.

These analytical methods provide elemental composition of solids and liquids as well as structural parameters of powders, thin films and bulk materials.

APD 2000 PRO is a Theta/Theta powder diffraction system for XRD qualitative and quantitative analysis of polycrystalline materials.

With an achievable peak resolution $< 0.04^\circ 2\theta$ and an angular accuracy within 0.02° over the whole 2θ angular range, **APD 2000 PRO** guarantees the necessary level of performance for the most demanding X-ray diffraction material investigation in a low cost configuration.

Which Measurements can be performed with APD 2000 PRO?

- Qualitative and Quantitative Crystalline Phase Analysis
- Degree of Crystallinity Calculation
- Polymorph Screening
- Cell parameters, Crystallite size and Lattice strain determination
- Rietveld refinement for structural characterization

APD 2000 PRO X-Ray Diffractometer Features

APD 2000 PRO, offers characteristics and options that provide maximum flexibility without quality compromises and with reliable data analysis.

Theta/Theta Goniometer: GNR has developed a compact goniometer, adopting stepper motors with optical encoders, to ensure extremely precise angular positioning.

Optics: X-ray beam collimation is obtained by variable slits that guarantee a perfect alignment of the beam in the equatorial plane, while in the axial plane the divergence is limited by interchangeable Soller slits.

APD 2000 PRO can mount glass or ceramic X-ray tubes with high reproducibility and stability of focus position.

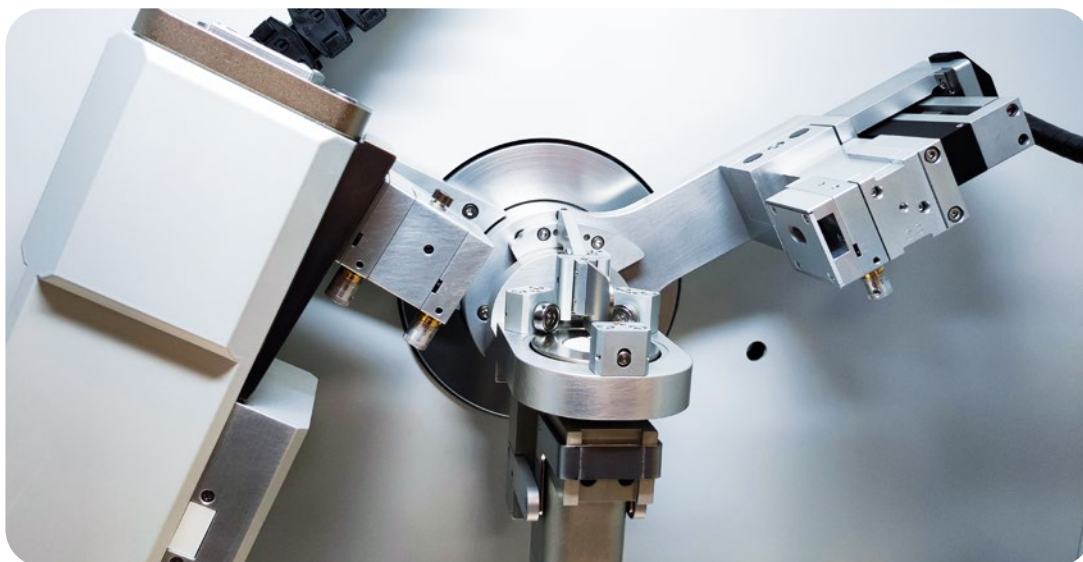
X-ray Detectors: Silicon Drift Detectors (SDDs), multi strip linear and area detectors (1D, 2D).

Sample holders: several sample holders are available to meet the specific needs of each laboratory (rotating sample stage, standard and custom sample holders, air sensitive sample holder, spinning 6 position multisample holders).

APD 2000 PRO

Theta/Theta Goniometer

The Theta/Theta configuration allows easy sample preparation, as the measured surface remains in a horizontal plane while the tube and the detector arms are rotating. This configuration is equally suitable for the analysis of loose powders, liquids, coatings and heavy samples that cannot be easily fixed in position.

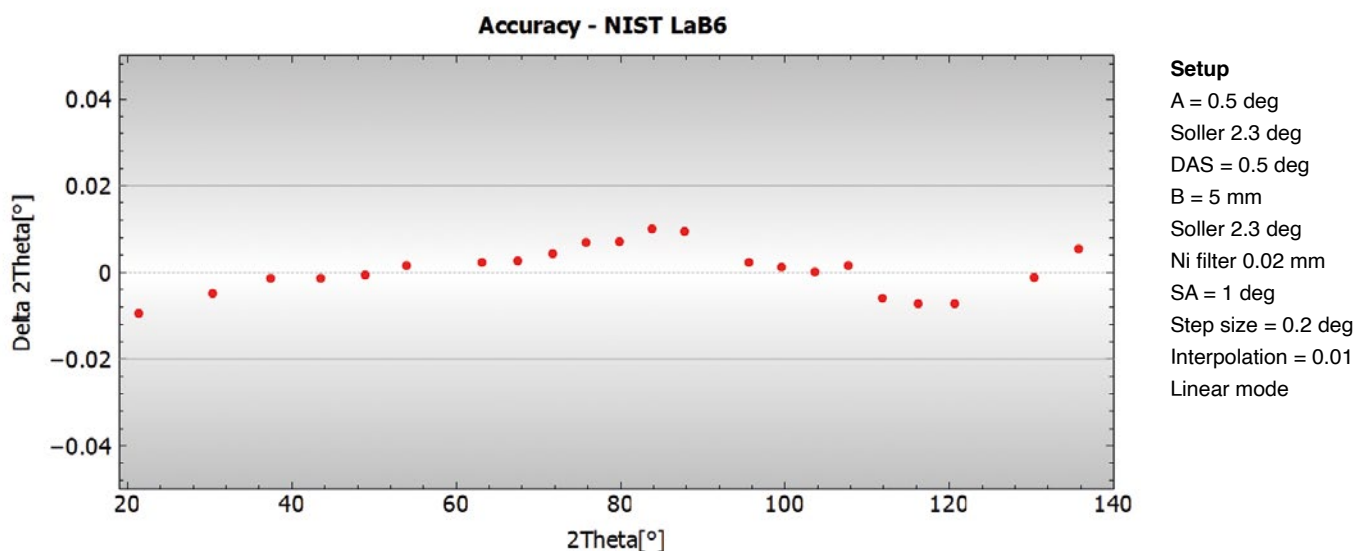


High-precision goniometer

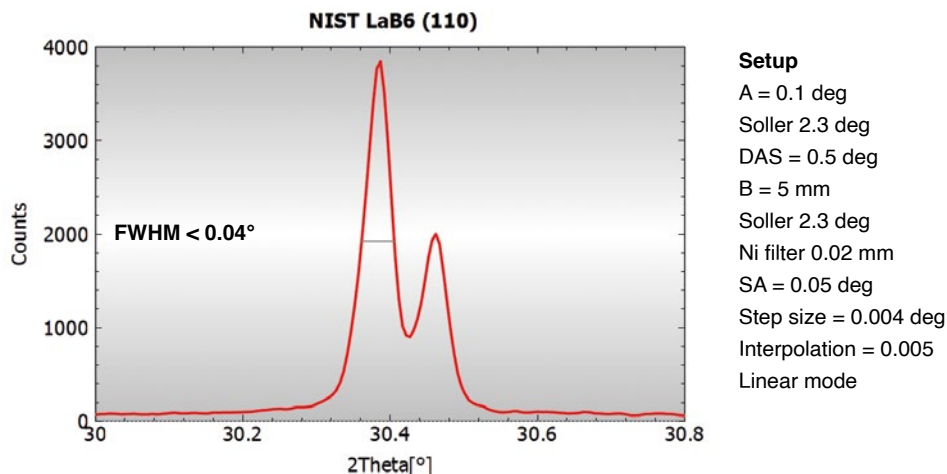
Goniometer Accuracy

GNR developed a high performance goniometer featuring high precision and outstanding results.

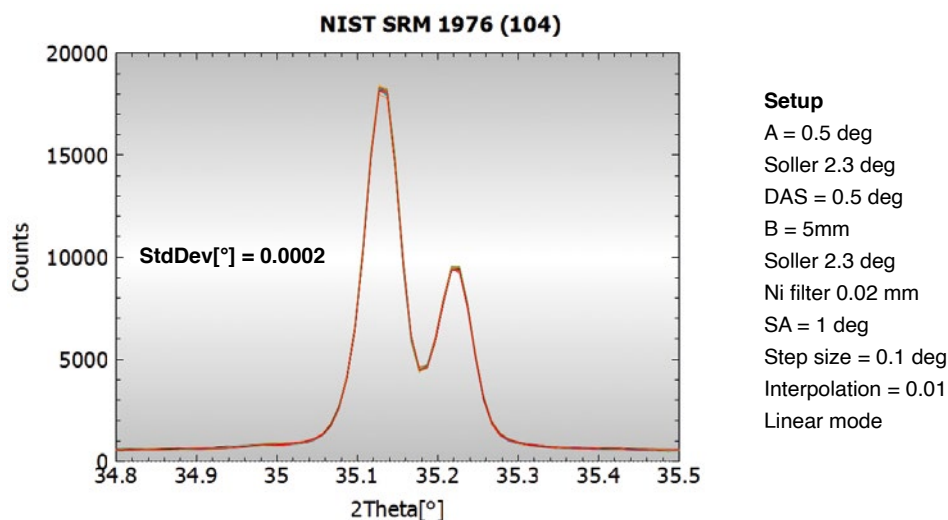
An angular accuracy better than $\pm 0.02^\circ$ over the whole 2-Theta range guarantees optimal instrument alignment to support accurate and reliable analysis.



Goniometer Resolution



Goniometer Reproducibility



25 sequential measurements with Corundum 1976 (104) peak

A = divergence slit DAS = divergent beam anti-scatter slit – B = anti-scatter slit

APD 2000 PRO is pre-aligned at delivery. Every single instrument must pass strict internal test procedures based on the internationally accepted NIST Standard Reference Materials (SRM) 1976 (Alumina), 660 (Lanthanum Hexaboride) or 640 (Silicon).

Safety Assurance

APD 2000 PRO complies with the complex statutory requirements regarding X-ray, machine and electrical safety. Maximum X-ray safety with radiation level significantly below the annual dose limit for general public (1 mSv/year). The radiation enclosure door cannot be opened when X-rays are on and the system immediately switches off if shutter is forced to open. This function completely protects users from radiation exposure.

Dectris 1D Hybrid Photon Counting (HPC) microstrip detector

Dectris multi strip detector takes full advantage of both Hybrid Photon Counting (HPC) and Single Photon Counting technologies, providing:

- X-ray direct detection;
- noise-free performances;
- high intensity measurements;

Our Dectris 1D detector allows to perform data collection in an extremely short time: it can capture simultaneously a large angular range, thus reducing measurement time from hours to minutes.

Dectris 1D detector can be used in 0D-mode and turned by 90° to cover an extremely large dynamic range.



Dectris 1D

Main Specifications	Dectris 1D	Main Specifications	Dectris 1D
Number of counting pixels	640	Dynamic count rate range [counts/s]	Up to 1×10^{10}
Pixel size [μm]	50	Energy range [keV]	5-40
Active area [mm^2]	256	Cooling	Air

Advacam MiniPix-TPX3 2D solid-state hybrid pixel detector

Advacam 2D detector is position, energy and time sensitive. For each ionizing particle (e.g. X-ray photon) it digitally registers its position, energy, time of arrival and track shape. The information on each detected particle is either read-out immediately at maximum rate of 2.3 million hit pixels per second.

Main Specifications	Advacam MiniPix-TPX3
Number of counting pixels	256 x 256
Pixel size [μm]	55 x 55
Active area [mm]	14 x 14
Acquisition mode	0,1 and 2D



Advacam MiniPix-TPX3

Silicon Drift Detectors (SDDs)

Silicon Drift Detectors (SDDs) combine a large sensitive area with a small value of the output capacitance and are therefore well suited for high resolution, high count rate X-ray spectroscopy like EDXRF.

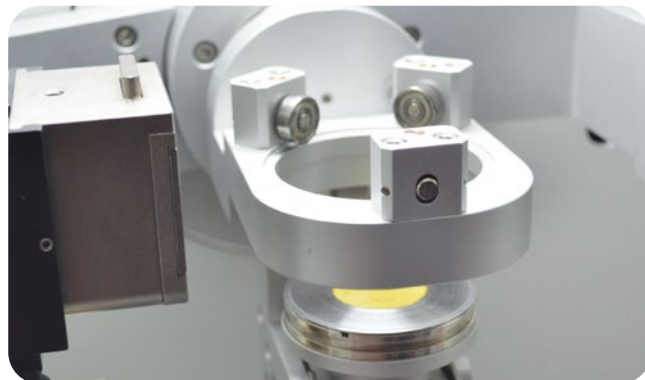
Main Specifications	Silicon Drift Detector - SDD
Active area	10 – 170 mm^2
Energy resolution	Shaping time 1 μs : $\leq 133 \text{ eV FWHM@Mn K}\alpha$
Cooling	Air



Silicon Drift Detector

Rotating sample stage

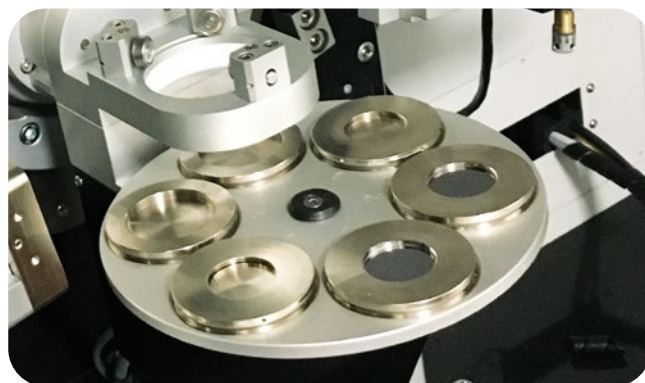
Rotating sample stage enhances measurement quality for sample with non-randomly oriented crystallites.



Rotating sample stage

Spinning multisample holder

Automatic sample positioning for long-time analysis without user attendance.



Spinning multisample holder

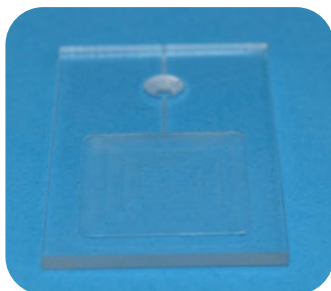
Specimen holders

GNR provides several dedicated solutions for different kinds of specimen. These fit the analytical requirements and guarantee the best achievable data quality. Here below an overview of some specimen holders available. Upon request tailored solutions can be realized to further optimize the analytical results.

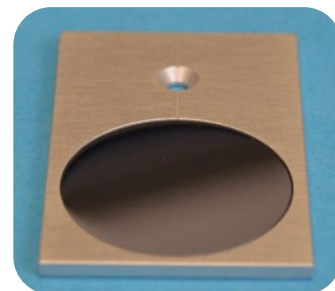
Flat specimen holders (length 44 mm)



Front loading sample holder
Material: Al
Cavity:
HxL: 20 mm x 20 mm
Depth: 1 mm

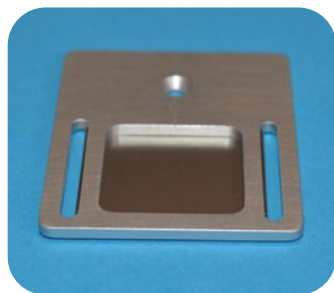


Front loading sample holder
Material: PMMA
Cavity:
HxL: 20 mm x 20 mm
Depth: 0.2/0.5 mm

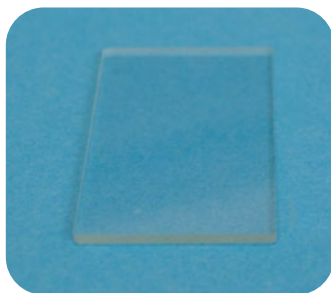


Si zero background sample holder
Material: Al
Cavity:
Diameter: 24.7 mm
Depth: 0.5/0.1 mm

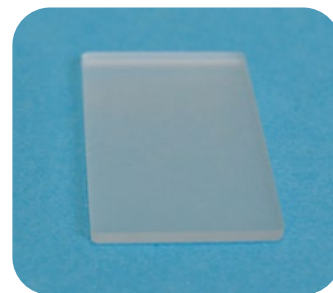
Sample Holders



Back loading sample holder
Material: Al
Cavity:
HxL: 20 mm x 20 mm
Depth: 1.9 mm

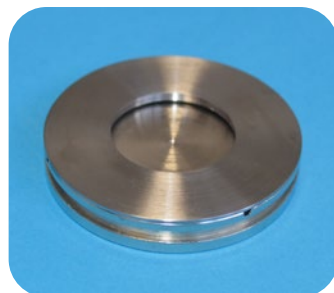


Glass slide for clay oriented mount

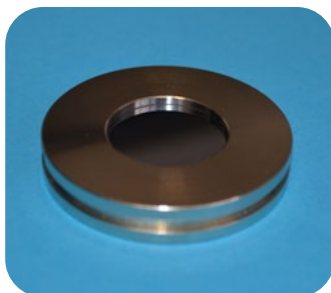


Glass slide for side loading sample holder filling

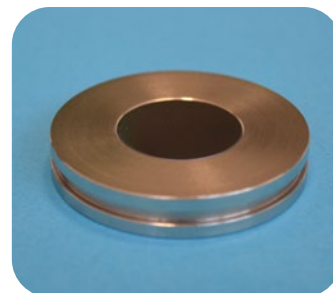
Circular sample holders (external diam: 51.5 mm - Herzog press)



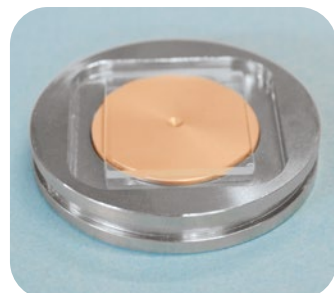
Back loading sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 2.5 mm



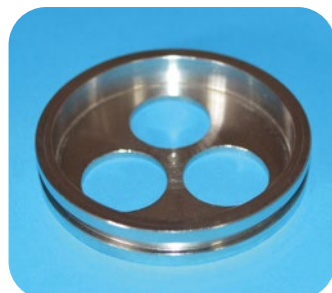
Side loading sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 2 mm



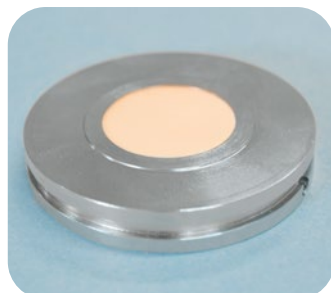
Si zero background sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 0.2 mm



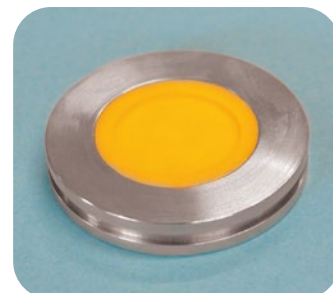
Clay oriented aggregate mount sample holder
Material: steel and glass
Cavity:
Area: 37 x 37 mm²
Depth: 2.5 mm
Glass: 25x25 mm²



Massive sample holder
Material: steel
Cavity:
Diameter: 45 mm
Depth: 7 mm



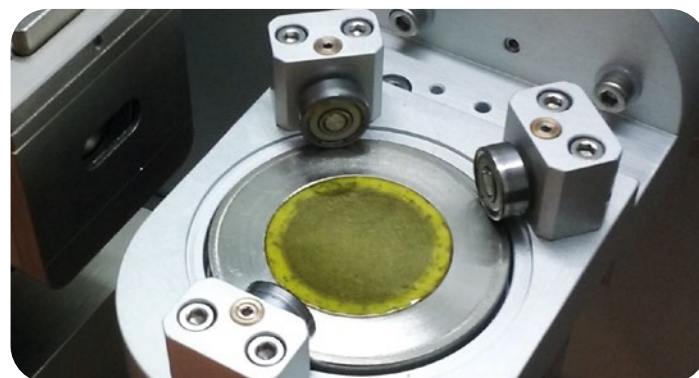
Air monitoring filter sample holder
Material: steel
Cavity:
Diameter: 25.7 mm



Air sensitive sample holder
Material: steel and teflon
Cavity:
Diameter: 25 mm
Depth: 1.5 mm
Foil: Kapton, 8 µm



Massive sample holder

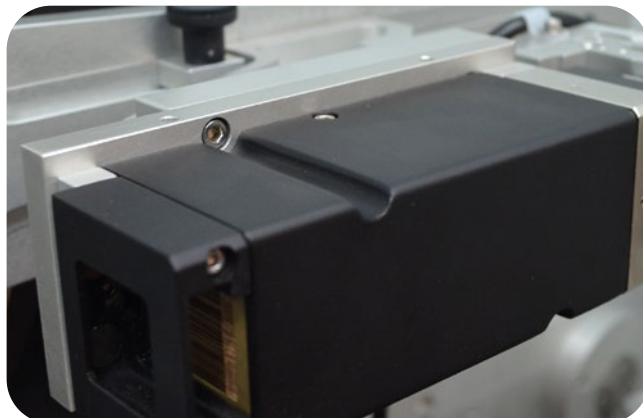


Air sensitive sample holder

Long Equatorial Soller Slit (LESS)

The Long Equatorial Soller Slit (LESS) consists of a set of parallel plates separated by equal spacers, which results in a set of narrow parallel collimators defining the equatorial acceptance angle seen by the detector.

The LESS attachment is used in the **APD 2000 PRO** diffractometer when polycrystalline samples in the form of coating have to be analysed with the grazing incidence geometry.



Long Equatorial Soller Slit (LESS)

Non-ambient X-Ray Diffraction

Non-ambient X-ray diffraction has become an indispensable technique to understand the influence of temperature, atmosphere or pressure on materials of any kind. Besides its relevance for conducting research, this knowledge is essential for optimizing technical processes and performing quality control in industrial applications.

The control electronics is integrated in the heating stages and provides easy operation.



BTS 500

BTS 150/500

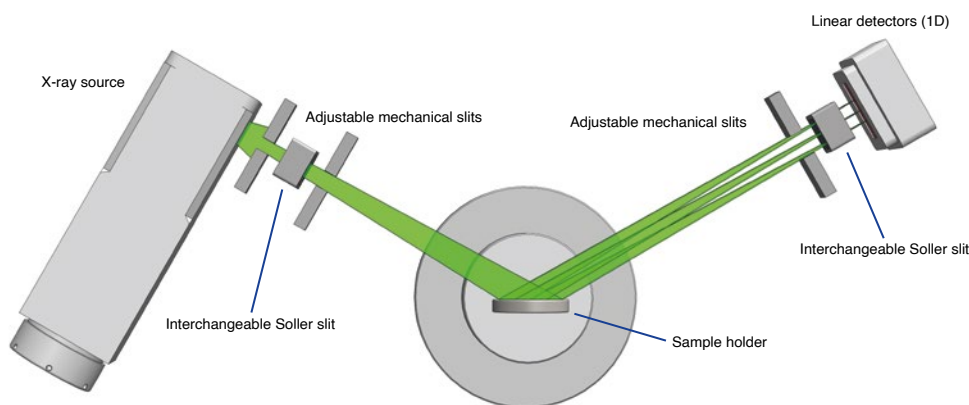
Technical data

Temperature range	-10 °C to 150 °C (BTS 150)
	Ambient to 500 °C (BTS 500)
Atmospheres	Air, inert gas, vacuum (10^{-1} mbar)
X-ray geometry	Reflection

Phase analysis is the study of the different polycrystalline materials within a sample.

One phase is identified among the others due to its unique powder diffraction pattern which arises from its unique combination of composition and crystal structure.

The analysis is applicable to all types of crystalline materials and can be restricted to identification only or extended to full quantitative analysis.



EXPLORE POWDERS

- Crystalline phases ratio (E. Calibration, RIR, Rietveld)
- Amorphous content in samples
- Crystal structure
- Phase transitions
- Crystallinity index and percentage

GNR APD 2000 PRO fits well customer analytical needs in many fields:

- | | | |
|--------------------------|-------------------------|----------------------|
| ▪ Geology and Mineralogy | ▪ Catalysts | ▪ Chemicals |
| ▪ Clays | ▪ Polymers | ▪ Pharmaceuticals |
| ▪ Glass - Ceramics | ▪ Forensics | ▪ Cosmetics |
| ▪ Cements | ▪ Agricultural Sciences | ▪ Environmental |
| ▪ Petrochemicals | ▪ Biosciences | ▪ Art and Archeology |



Geology and Mineralogy
Determine the composition of raw material



Petrochemicals
Analysis of solids obtained during the drilling process



Chemicals
Characterize unknown materials and support R&D



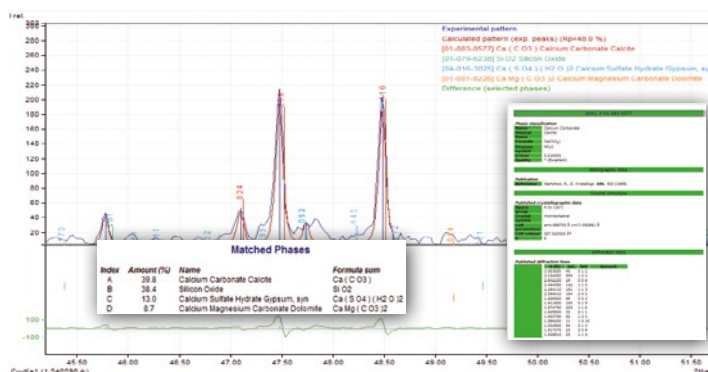
Pharmaceuticals
Quality control, identification and polymorphs screening

Phase Identification and Quantitative - Mineralogy

GNR APD 2000 PRO can be used for phase identification and quantitative analysis in mineralogy domain.

Match! can identify the phases present in the sample and report the weight percent composition.

In this case the sample is a mixture of Quartz, Calcite, Gypsum and Dolomite.

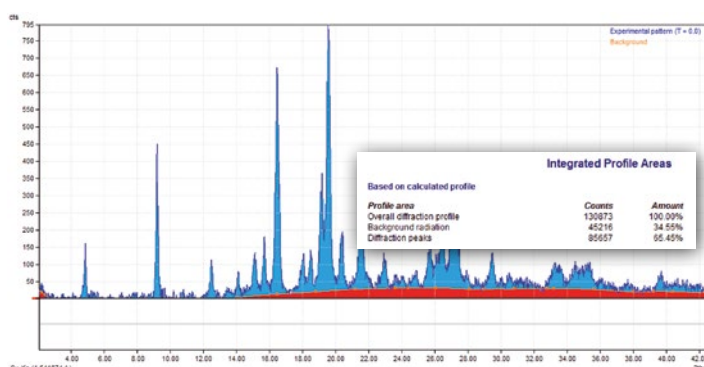


Quantitative Phase Analysis using Match!

Crystallinity Degree Calculation - Pharmaceutical

In pharmaceutical industry product development and quality assurance steps, it is common to measure the amorphous phase amount in order to determine the degree of crystallinity.

It is important to monitor the amount of the amorphous phase within a drug because of its thermodynamic instability, relative to the crystalline state. Match! allows to determine it easily through the Degree of Crystallinity calculation.

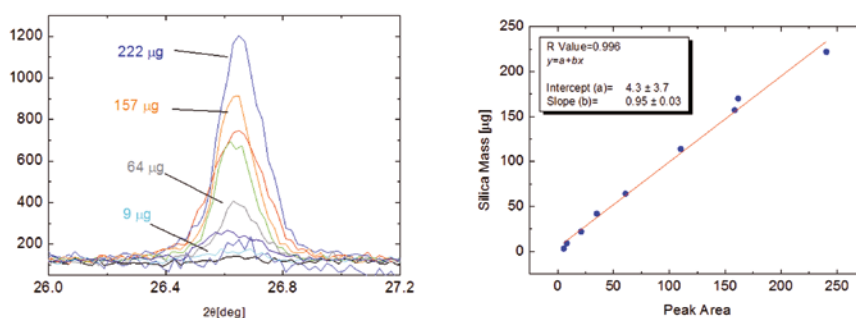


Crystallinity Degree Calculation using Match!

Silica Dust Monitoring - Environmental

Crystalline silica is a basic component of soil, sand, granite, and many other minerals. Quartz is the most common form of crystalline silica. The seriousness of the health hazards associated with silica exposure is demonstrated by the fatalities and disabling illnesses that continue to occur in sandblasters and rock drillers. Crystalline silica has been classified as a human lung carcinogen. Measuring and quantifying silica dust collected on air filter is important to protect health of workers involved in activity exposed to silica dust.

Quartz (101) diffraction peak area is proportional to the mass of silica dust deposited on a filter. Using NIST – SRM 1878 quartz standard is possible to calculate a calibration curve for silica dust mass quantification and monitoring.

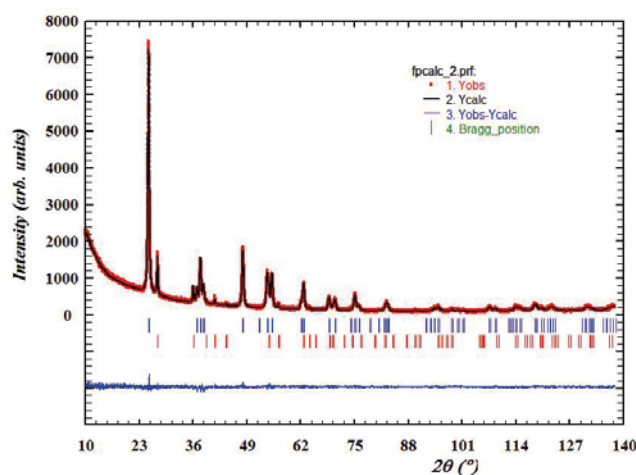


Calibration curve for silica dust mass quantification

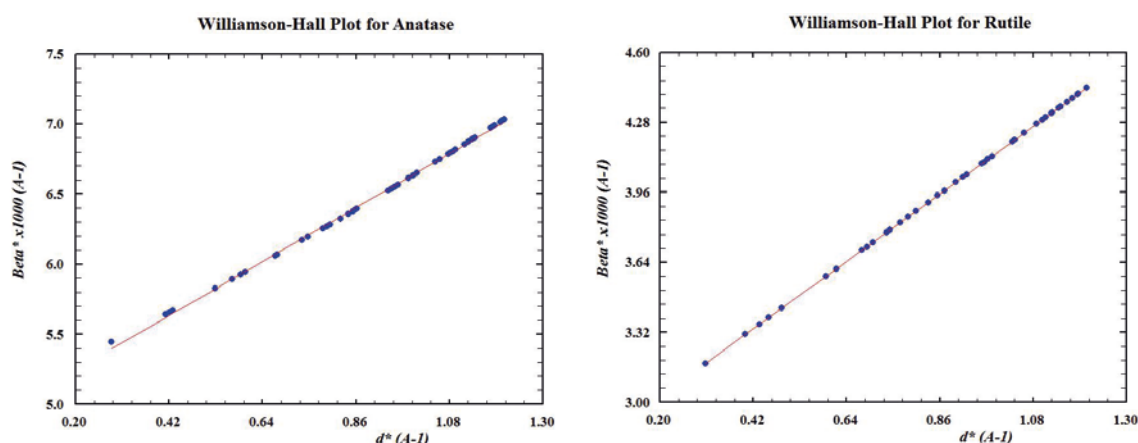
Crystallite size estimation of nanopowders

Nanocrystalline materials are currently an active field of research: the possibility of tailoring their functional properties, by modifying their microstructure, makes them very appealing. XRD is an effective technique to study their defective microstructure through Line Profile Analysis: **APD 2000 PRO** powder X-ray diffractometer equipped with Dectris linear detector allows to perform Rietveld refinement with extraction of microstructural parameters in a fast and easy way. After instrumental broadening determination by means of NIST SRM 660 (LaB6), a nanocrystalline mixture of Rutile and Anatase (NIST 1898) was analysed and apparent average crystallite size for both phases was estimated by simple Scherrer formula (MATCH!) and integral breadth methods implemented in FullProf Rietveld program¹.

¹J. Rodríguez Carvajal and T. Roisnel. Materials Science Forum 443-444, 123-126 (2004).



Rietveld refinement plot for Anatase (blue) and Rutile (red) mixture.



W-H plots for Anatase and Rutile. Lorentz-Lorentz approximation is assumed

Apparent crystallite size [nm]				
Phase	Peak	Match!	Match! Uncorrected	Certificate Information Value
Anatase	(200)	23.6	23.3	23.6
Rutile	(111)	48.6	39.3	44.1

Results of Scherrer analysis for selected peaks

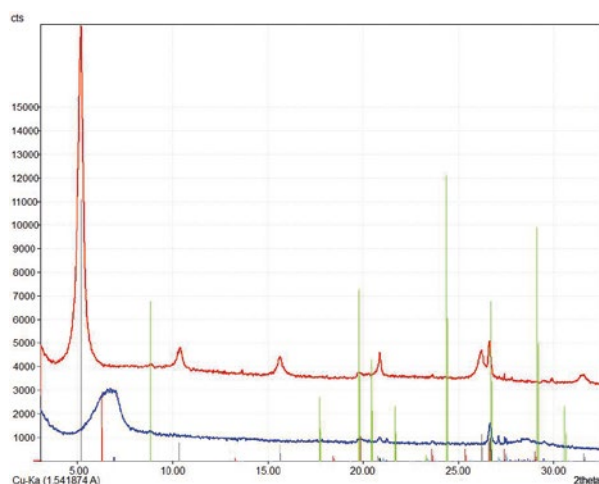
Volume weighted apparent crystallite size [nm]			
Phase	FullProf	W-H Plot	Certificate Information Value
Anatase	20	20	19±2
Rutile	36	36	37±6

Results from FullProf microstructural analysis

Oriented aggregate mounts for Clay minerals identification

The correct identification of clay minerals is very important¹: the clay fraction is separated from the sample and prepared as an oriented aggregate on glass slides to enhance diagnostic basal reflections signal. The corresponding d-spacing contraction or expansion during treatments allows a clear identification, especially if layer interstratification is present. Typical treatments are: air drying, glycolation with ethylene glycol, heating to 400°C and 550°C. Diffraction pattern collection must start at low angles ($2\theta < 5$ deg.) in order to collect basal reflections at very large d-spacings (e.g. Smectite, Chlorite groups)².

In the example reported below a Bentonite sample (SWy-2) was deposited directly as an oriented aggregate on a glass slide. First air drying and then ethylene glycol treatments were performed. Diffraction patterns were collected from 2θ 3 to 35 deg by **GNR APD 2000 PRO** diffractometer with Cu FF anode, Ni filter and Dectris 1D mod. Advanced 640 linear detector.



The blue pattern represents the air dried sample: the red vertical bar corresponds to Na-Montmorillonite phase 001 peak ($d \sim 14$ Å) with different water layers (1 to 2), while the green one the Illite 001 ($d \sim 10$ Å). Quartz (101) peak is visible at 26.64 deg, while Feldspars are detected in 27.1-27.5 deg region.

The clear difference after glycolation (red pattern) is the enhanced signal of the Montmorillonite basal peak and its shift to larger d values (~ 17 Å), which is compatible with a glycolated-Montmorillonite clay mineral (grey bar), according to ICDD PDF4 and USGS Clay Mineral Identification Flow Diagram³. Higher orders (002, 003, 004, 005 and 006) are visible now too. Illite is unaffected by glycolation, as expected.

¹ F. Bergaya, B.K.G. Theng and G. Lagaly, Handbook of Clay Science, Developments in Clay Science, Vol. 1 2006 Elsevier.

² A laboratory Manual for X-ray Powder Diffraction, USGS, <http://pubs.usgs.gov/of/2001/of01-041/>

³ <https://pubs.usgs.gov/of/2001/of01-041/html/docs/flow/start.htm>

Performances at low scattering angle: Silver behenate

Configuration:

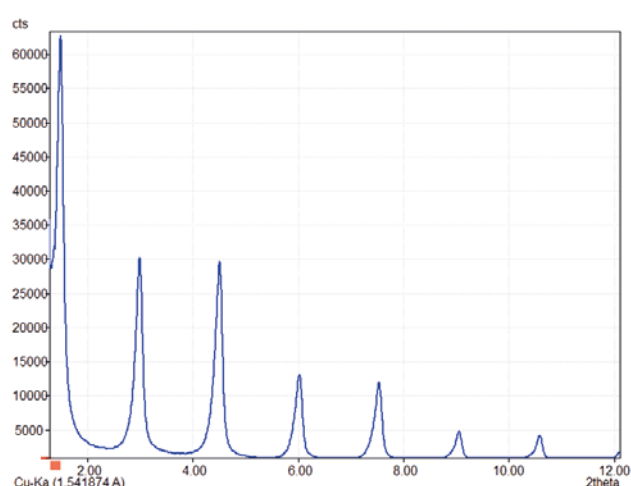
Divergence slit = 0.5 deg

Anti scatter slit = 0.5 deg

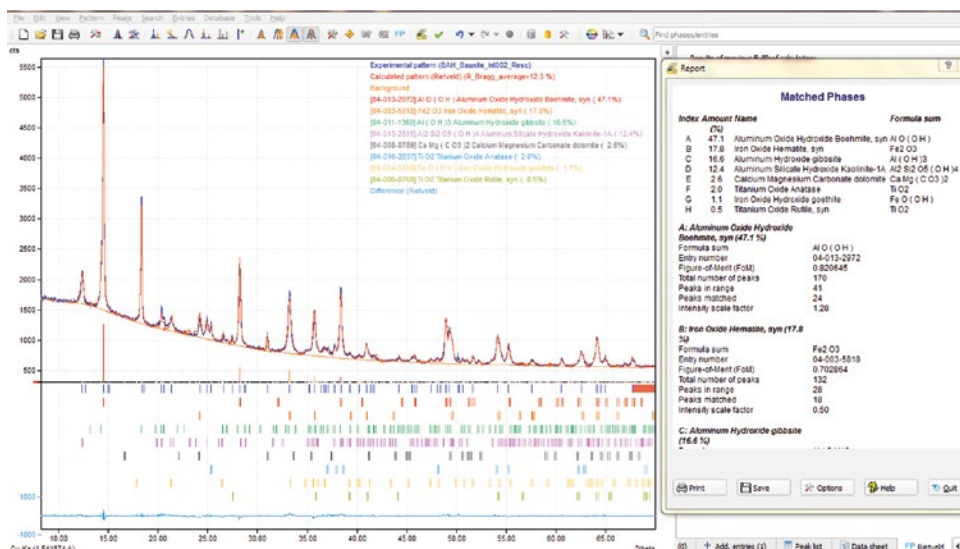
Soller slits = 2.3 deg

Detector slit = 2.2 mm

Dectris 1D mod. Advanced Active area = 0.8 deg



As reference database, you can apply the included free-of-charge COD database and/or ICSD/Retrieve (if you have a valid license), use any ICDD PDF product, and/or create a user database based on your own diffraction patterns.



Configurations	Theta/Theta geometry
Measuring circle radius	160 mm
Scanning angular range	- 5° < 2-Theta < + 160° (depends on accessories)
Angle positioning	Stepper motors with optical encoders
Angular accuracy	Better than ± 0.02° over the whole 2-Theta range
Angular resolution	< 0.04 2-Theta on LaB ₆ (110) peak
X-ray generator	3 kW (option: 1.2 kW: 40 kV / 30 mA - with internal water chiller)
Max. output voltage	60 kV
Max. output current	60 mA
X-ray tube	Glass (option ceramic), Cu anode (options: Co, Fe, Cr, Mo, W, Ag)
Focus	0.4x8 mm FF (Fine Focus) (options: 0.4x12 mm LFF; 1x10 mm NF)
Divergence slit	Variable 0-6 mm
Anti-scatter slit	Variable 0-6 mm
Receiving slit	Variable 0-14 mm
Interchangeable Soller slits	2.3° (standard) (options: 1°, 2°, 4°)
Detectors	1D/2D detectors Silicon Drift Detectors (SDDs)
External dimensions	900 W x 793 D x 1583 H mm
Weight	312 Kg
Water cooling supply (without optional water chiller)	Min. flow rate: 4l/min – Pressure 4 bar to 6 bar
Maximum power consumption (including water chiller)	10 kVA

